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Continuous Vacuum Drying of Whole Milk Foam. III. Optimization Operations

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Abstract

A process for vacuum foam drying whole milk has been investigated. Preliminary work was done on mechanical modifications, development of techniques and instrumentation, and identification of the most important control variables. This paper discusses the subsequent phase of experimentation, wherein a three-year study was made, utilizing experimental designs and mathematical model simulation. The objective of the study was to determine operating conditions which would permit year-round operation, in spite of seasonal variation in milk foaming characteristics, at an economically desirable rate, with good product attributes. Conditions were found and tested in the pilot plant. Although no test has been made on highly stable (slow-drying) milk, the mathematical predictions were largely borne out in operation. The process was scaled up by two-thirds, and 42 trials were made during 4.5 months. Moisture and 5-hydroxymethylfurfural contents were consistent and adequately low.

Introduction

An extensive research program has been conducted at the Eastern Utilization Research and Development Division to develop an economically feasible process for the production of vacuum foam-dried whole milk. This product has unique properties of reconstitution, dispersing rapidly in ice water. It has very good initial flavor, and largely retains both characteristics for more than one year at 4 C (2). The milk has been produced experimentally on a continuous basis with acceptably good flavor, adequately low moisture level, with low heat damage and a marginal rate from an economic standpoint (1, 13).

Drying efficiency in the process is dependent on the extent of foam formation during drying and on foam stability. It would probably not be feasible to monitor foam stability during production. These characteristics vary seasonally, so that acceptable output could only be achieved during some seasons of the year. It was found, generally, that foams from milk produced in the late fall and winter were less stable than those from late spring and summer, and dried to a lower moisture content, or at a higher rate. The stable summer foams dried at an uneconomic rate. Holden et al. (12) developed a test to characterize these phenomena in the milk used for drying studies. Schoppet et al. (13) concluded that the remaining problem in achieving an acceptable process for the continuous vacuum foam drying of whole milk was to optimize the process, while simultaneously achieving year-round operation.

Before discovery of the relevance of seasonal variation in the raw material, the requisite equipment had been developed for the process, and insight gained into those parameters which influenced drying rate and product quality. With the groundwork thus laid, it was decided to study the process using a stochastic model, applying statistical design techniques, and mathematical model simulation. It was hoped that a mathematical model of the process would allow an unambiguous optimization, and would permit study of the seasonal variation. It was further hoped that operating conditions could be found which would allow economically feasible year-round operation. As a matter of philosophy and practicality, it was decided that efforts should be directed toward determining one set of operating conditions, rather than a different set of conditions for each season of the year. This paper presents the experimentation and mathematical studies employed in this program.

Experimental Procedure

Preface. In the preliminary stages of this program, 13 variables were identified which, it was believed, had a significant effect on product rate or quality. In many cases, these variables were not directly recorded during experimentation but, rather, calculated from the raw data.

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Table 1 shows all these model parameters in their final form. It should be recognized that their form was altered from time to time, as a new form was found to be more relevant, or subject to more accurate determination. Those parameters which required considerable alteration were:

Solids content of the concentrate, X_1. It was found that dryer performance on a given day is directly related to concentrate viscosity, which is dependent on concentrate solids, fat content, and other characteristics of the fluid milk. However, the viscosity-solids relationship has a marked seasonal character, and a better over-all model was found by using concentrate solids rather than viscosity as X_1. For operating conditions, viscosity was then estimated from the predicted solids and fat content, taking season into account.

Belt loading, X_4. The width of the foam ribbon deposited on the belt is directly dependent on feed rate. However, it also varies somewhat with viscosity, gas content, etc. To achieve sufficient precision, the foam ribbon was photographed, ribbon width estimated from the known belt width, and belt loading then calculated from the belt speed, feed rate, and ribbon width.

Fat content, X_{12}. It was intended that all studies would be made at 26% milk fat. In practice, variations in fat content had a marked effect on drying efficiency. In the earlier phases of experimentation, pre-run adjustment of fat content was not sufficiently precise to fix this variable (analyzed in the dry product). Consequently, it was necessary to include the variable in the model.

Foam time, X_{13}. To characterize the seasonal foam behavior of the raw material, the test described by Holden et al. (12) was developed. This test resulted in an R_{fs} value, i.e., rate of foam subsidence, describing how rapidly a test foam collapsed after being formed by an inert gas. As a broader base of data accrued, it became evident that this description was inadequate. The height to which the foam expands before collapsing is equally relevant. Therefore, X_{13} was redefined as foam time, or initial foam height divided by R_{fs}, giving the time required for a specific foam to collapse.

The dependent (response) variables considered to describe process efficiency and product quality were:

1. Product rate (calculated).
2. Product moisture (standard toluene extraction).
3. 5-Hydroxymethylfurfural (HMF) content (9).
4. Solubility index (American Dry Milk Institute Test).
5. Product dispersibility (15), with the modification that a wrist-action shaker was used, for the same time interval, instead of hand stirring.
6. Bulk density (tamped).
7. Flavor (2).

Preliminary factorial design studies established the following:

1. Only one variable of those considered could be regarded as insignificant in differential effect over the range studied: the heat level on the upper (product) side of the belt after the nozzle (X_8). This was held constant for the remainder of these studies.
2. A significant number of variables interacted with one another and were individually nonlinear in their effect. Hence, designs utilized could not be those resulting in linear models, but three-level factorial, at least.
3. Study of the foaming characteristics of the

TABLE 1. Variables studied.

X_1	Concentrate solids content	Per cent
X_2	Dryer feed concentrate gas content	Cm^3 gas/liter ungassed concentrate
X_3	Lecithin content	Grams lecithin/g solids
X_4	Belt loading	Pounds concentrate (W.B.)/ft^2 (kg/m^2)
X_5	Chamber pressure	Millimeters Hg absolute
X_6	Nozzle aperture	Millimeters
X_7	Residence time (from nozzle to cold drum)	Minutes
X_8	Calrod temperature, first zone, product side[a]	C
X_9	Hot drum temperature	C
X_{10}	Calrod temperature, first zone, belt side	C
X_{11}	Nozzle temperature	C
X_{12}	Fat content	Per cent milk fat
X_{13}	Foam time (seasonal parameter)	Minutes

[a] Held constant in final analysis.

milk revealed that not only did the milk vary significantly day-to-day in a random manner, but it exhibited a seasonal trend in the fall and spring. During winter and summer, no long-term trend in foaming was discernible.

4. A response surface approach, using Box-Wilson rotatable orthogonal designs (3), based on factorial or fractional factorial designs (5, 8), was chosen. However, a design based on one-fourth (2^{12}), a reasonable choice, would have taken far too long, at an average rate of one experimental point per day (later raised to 1.5 points per day). In addition, as seasonal variation was not subject to control, it was necessary to restrict a design to one season in length. For these reasons it was decided to utilize experimental designs based on a smaller number of variables in any given season, and build up a model piece by piece. The obvious time restrictions imposed the additional requirement that each design be separately amenable to analysis, so that the next design could be placed closer to optimal conditions.
5. Of the seven dependent variables, product rate is dependent only on belt loading and residence time, considering concentrate solids as approximately constant. The rest presumably were affected by most or all of the variables studied. Each quality response was defined in terms of "less than or equal to" a given level. As a working hypothesis, it was tentatively concluded that, for a given product rate, if the moisture and hydroxymethylfurfural constraints were satisfied, all other quality measures would also occur at acceptable levels. It was believed that holding heat damage to a sufficiently low level would largely ensure a good quality product.

Experimental approach. The process during this period conformed to the flow sheet offered by Schoppet et al. (14).

Mathematical models were required for moisture and HMF contents. The models used were quadratic response surfaces, expressed in general as:

$$Y = a_{oo} + \sum_{i=1}^{12} a_{oi}X_i + \sum_{i=1}^{12} \sum_{j=i}^{12} a_{ij}X_iX_j$$

As stated above, it was considered that the required coefficients could be estimated, one group at a time, within seasons. However, the effect of the uncontrolled seasonal variable, X_{13}, could not be dealt with in this manner, as no seasonal effects could be seen in summer or winter. Likewise, the interactions with X_{13} could be estimated only when X_{13} was changing appreciably. Consequently, two forms of design were employed. During "constant" seasons (winter and summer), the controlled variables were studied, using designs based on fractions of 2^n factorial designs, with composite and center points for the second-degree terms. During "transition" seasons (spring and fall), when a large change in foaming behavior could be anticipated, fractions of 3^n factorial designs were used, blocked so that the blocks were approximately confounded with seasonal change. The controlled variables studied were not confounded with the seasonal change. These designs were not treated as 3^n designs for analysis, but the three levels provided information for nonlinear terms when analyzed by multiple regression techniques. In addition, of course, some information was gained from the comparison of experimental designs run in summer versus winter. Table 2 summarizes the experiments run and the type. It should be noted that "add-on" studies were occasionally performed. After a design was completed, one or more of the design variables were held constant and a new variable, previously held constant, substituted into the design. By this mechanism, information could be obtained on the new variable and its interactions with those still "active." As half of this design would have been previously completed, it was an economical means of obtaining some additional data in a limited time.

Not listed in Table 2 are four designs run in 1962 and 1963, used to establish the relative effects of the variables, their forms, and the likelihood of their significance. They were not used

TABLE 2. Experiments and variables studied.

From	To	Variables	Type
11/63	12/63	1,3,4,5,13	T
1/64	2/64	1,4,7,9,10	C
1/64	2/64	3	A
3/64	5/64	1,2,3,4,13	T
7/64	10/64	4,5,6,7,9,10	C
11/64	12/64	1,3,7,13	T
1/65	2/65	2,3,5,6,11	C
4/65	6/65	1,5,6,11,13	T
8/65	10/65	2,4,7,9,10,11	C
11/65	4/66	2,4,5,7,10,11	C
11/65	4/66	9	A

Code: T, Transition design—based on 3^n.
C, Composite design—based on 2^n.
A, Add-on design.

in the subsequent analysis, as daily determination of foam stability was not instituted until fall, 1963.

Two variables, heat levels above and below the belt after the hot drum, were found to be of secondary significance and, in fact, were not studied systematically beyond this point.

Overall, the experiments amounted to slightly more than one phase of evolution. With each design, a very conservative move was made toward the presumed optimum. The danger in this approach is that information not then available on a variable might actually indicate a move opposed to the one made. Fortunately, this did not happen. As each design was completed, the results were added to those previously obtained, and the entire data set analyzed by nonlinear multiple regression. While it was possible to analyze these experimental designs as simple factorials, the information thus gained was used only in preliminary estimates.

Some controls, critical to successful operation, but either not amenable to variation or believed not to be directly related to dryer output, were held constant through all experiments. These are listed in Table 3.

Analysis

As noted above, factorial analysis was used for each design, but only to acquire preliminary information. As each design was finished, the data acquired were added in and the cumulative data set reanalyzed, including each coefficient which had been studied in any design to that point. The significance of each full set, beginning in early 1965, was determined by analysis of variance. The models all exceeded the 95% confidence level ($P < .05$). As no marked saving in subsequent analyses would have resulted, and the determination would have been quite costly, no attempt was made to evaluate the significance of individual coefficients. From external knowledge of replication errors, which was believed applicable, it appeared that not only was the model significant but a lack of fit term probably was as well. However, no means was available to analyze a model more complex than the quadratic and theoretical considerations did not offer any useful transformations. Consequently, it was assumed that the quadratic model would be close enough.

Each model required the formation of the least-squares normal equations from the data, and solution of 90 simultaneous equations (7). This was done for moisture and hydroxymethylfurfural content responses. Some early attempts were made to utilize solubility index in a model, but a poor fit was obtained. Due to the lack of methodology for dealing with three responses, the solubility index model was not pursued.

The fraction of variation accounted for by the regression is an explanation of the value R^2, where:

$$R^2 = \frac{\text{Sum of squares due to regression}}{\text{Unconditional sum of squares of Y}} \quad (8)$$

This value was used to evaluate successive models. As each design after the first three was added, R^2 increased. Variable transformations and substitutions were evaluated by this means. The variable changes referred to in the preface were made on the assumption that a higher R^2 represented a more valid form, as well.

After an acceptable model for moisture and for HMF was achieved, the model was studied by ridge analysis, an optimization technique developed by Hoerl (11). Ridge analysis determines the location of extrema as a function of the radius of the experimental hypersphere. A series of radii substituted permit the specification of maxima and minima and their coordinates in a two-dimensional form. The current mathematical techniques are discussed more fully by Craig et al. (7). Briefly, the quadratic models were treated in the following manner:

1. Fat content, X_{12}, was made a constant at the equivalent of 26% milk fat by substitution.
2. A product rate was chosen, specifying the relation between belt loading and residence time. Liberty was taken here in assuming a constant concentrate solids and a constant ribbon width for calculating belt loading, rather than relating solids to X_1

TABLE 3. Conditions held constant during study.

1. Evaporator discharge temperature	51.7 C
2. Concentrate temperature at homogenizer	57.2 C
3. Concentrate homogenization (2-stage)	3000/500 psig (210.9/35.2 kg/cm²)
4. Votator speed	650 rpm
5. Temperature at votator discharge	0 to 2.8 C
6. Third zone product side heat (steam)	100 psig (7.0 kg/cm²)
7. Third zone belt side calrods	0 volt

and making ribbon width another dependent variable. The effect of this was that actual product rates were ±2%. This was checked in retrospect and did not significantly affect the location of the optimal operating point.

3. To minimize seasonal variation, solutions were constrained by substituting for two more independent variables, one for each dependent variable, using an equation which would minimize the maximum seasonal variation in moisture and HMF. X_{13}, the seasonal variable, was then dropped from the model.
4. The ridge analysis was performed at a series of belt loading values, each of which when held constant, also specified residence time for that product rate. Thus, six variables remained explicit.

In consequence, the models as analyzed were heavily constrained. It would not have been surprising if there were no solutions. In fact, feasible solutions were frequently found.

Most of the analyses were performed at 3.856 kg/hr product rate, which was relatively high. At the end of the experimental program, a solution (coded 88-2) was found at a nominal 4.082 kg/hr.

The last solution found at 3.856 kg/hr (84-1) and 88-2 were tested in the pilot plant over a four-month period, to determine their validity. In further mathematical work, by extrapolating the model beyond the design, solutions were found at 4.309 and 4.536 kg/hr.

In addition, it was considered possible that separate operating conditions for winter and summer might be desirable. Therefore, solutions were found wherein seasonal variation was not minimized, but rather values of X_{13} corresponding to summer and winter were substituted into the equations and the ridge analysis performed. This led to a set of operating conditions for each season.

Results

Table 4 presents the solutions found: 84-1, 88-2, 88-3 (4.309 kg/hr), 88-4 (4.536 kg/hr), 88-5 (summer), and 88-6 (winter). Points 84-1 and 88-2 were tested repetitively and both were close to the predicted value. 84-1 had a somewhat higher HMF content, while the moisture for 88-2 was lower than predicted. Output response at 4.309 kg/hr (88-3) was tested experimentally once, and the moisture content was again somewhat lower than predicted. Point 88-4 was estimated too late to check experimentally, and this should be remembered. Point 88-2 was chosen over 84-1, on the criteria of heat damage and output.

The experimental program was performed using a 20.32-cm stainless steel belt in the dryer, a 20.32-cm feed nozzle, and radiant heating rods to that scale. After testing the optimal

TABLE 4. Conditions.

	84-1	88-2	88-3	88-4	88-5	88-6
X_1	42.5[a]	38.0	36.6	34.0	25.1	23.5
X_2	200	205	208	217	200	199
X_3	.0840	.0840	.0836	.0769	.0860	.0860
X_4	.036	.037	.037	.037	.037	.037
	.176	.181	.181	.181	.181	.181
X_5	19.00	18.95	18.95	19.29	18.91	18.92
X_6	.83	.75	.73	.74	1.02	1.02
X_7	1.37	1.33	1.26	1.20	1.33	1.33
X_8	68.3	68.3	68.3	68.3	68.3	68.3
X_9	75.83	76.00	76.17	82.22	75.89	75.94
X_{10}	677.2	681.7	694.4	686.7	639.4	628.9
X_{11}	8.9	7.1	3.9	9.4	12.1	12.4
X_{12}	25.1[b]	25.1	25.1	25.1	25.1	25.1
X_{13}	......	......	......	......	22	8
			Predicted response			
H_2O	2.83	4.15	4.90	3.85	4.83	2.65
HMF	0	.7	1.5	.3	......	.5
Rate	3.9	4.1	4.3	4.5	4.1	4.1

[a] Viscosity, centistokes (40.5 C).
[b] Analyzed in dry product; equivalent to 26% milk fat in raw feed.

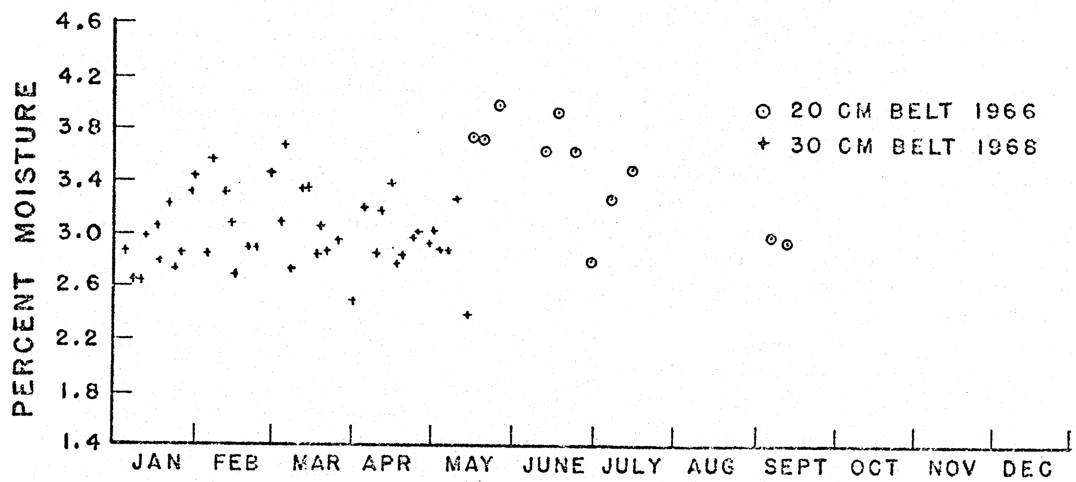

FIG. 1. Moisture content versus date of operation for repetitive studies.

conditions, output was scaled up from a nominal 4.309 kg/hr to a nominal 6.804 kg/hr by increasing belt width to 30.48 cm, and other equipment and flow rates proportionately. After reducing the difficulties engendered by scale-up, 42 runs were made, each identical to 88-2, within the limits imposed by control precision. Results of moisture analyses for these two groups of "routine" operations are presented in Figure 1. The season of January through May represents 30-cm belt data; beyond May, the 20-cm data taken before scale-up.

Discussion

The operating conditions chosen (88-2) demonstrated a fairly good prediction of dryer output. The scatter seen on Figure 1 is significantly greater than analytical error. It represents, primarily, variation in the control point. Twenty-centimeter belt data, while adequately close, appear to be a different population. This is most likely because scale-up was not exact in all ways (for example, dwell time in some heat exchangers was reduced). Unfortunately, while there was a substantial increase in foam stability in the summer, in 1961 through 1965, milk during the summers of 1966 and 1967 did not exhibit high foam stability. Acting on the assumption (in 1966) that feeding practices were responsible for the variation in foam stability, attempts were made to procure stable milk from outside the Eastern Pennsylvania-Delaware-Maryland-Virginia area. Milks from the Midwest and South were tried, without finding "summer" milk. The 6.8-kg/hr study was conducted in January through May, 1968. A check of foam stability in May showed that the milk still had "winter" characteristics. Thus, the conditions intended to permit operation with any foaming characteristics yet seen have been only tested for "spring" (or fall), "winter," and "deep winter," i.e., moderately stable, unstable, and quite unstable conditions. However, over this range of conditions, operation has been consistent and predictable. It may be conjectured, as feeding practices have changed since 1961, that "summer" milk is a result of fresh grass feed and may never be seen again in substantial quantities.

Conditions for 88-3 and 88-4 are as close to 88-2 as could be hoped. Obviously, they lie in the same region of the response surface, which allows a higher confidence in these extrapolated conditions. Points 88-5 and 88-6 are offered as a weak alternative to 88-2, in the event that it should not prove feasible. The predicted responses are not significantly better than 88-2. It should be noted that the responses given for the "all-season" conditions are central values, around which there will be a residual seasonal variation. Experimentally, 88-2 was found to vary $\pm$ 0.2% H_2O ($P \leq 0.05$), including control-induced variations.

The assumption made before the study, that other quality responses would be within acceptable limits if moisture and HMF content were, was largely correct. In the 42 repetitive runs made at 6.8 kg/hr, quality responses to drying were within acceptable limits.

It should not be concluded that the conditions found represent a maximum output for the process. A subsequent phase of study, at higher rates, would have been necessary to determine this. The constraint of time and the fact that 4.082 kg/hr (20-cm belt) was thought to be economically acceptable were the reasons that no higher rate was studied.

Conclusions

Acceptable operating conditions were derived from a mathematical model for the vacuum foam-dried milk process, which should allow operation over a wide range of milk foam stabilities. The conditions were found operationally feasible in the pilot plant, and production was successfully scaled up by two-thirds. The chief weakness of the experimental confirmation is that no markedly stable milks were encountered during the test periods of May 15 to July 15, 1966, and January 1 to May 15, 1968. However, all milk processed during these periods yielded an acceptable product under these conditions.

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